



SLOVENSKI STANDARD
SIST EN 12594:2007

01-julij-2007

BUXca Yý U
SIST EN 12594:2000

Bitumen in bitumenska veziva - Priprava preskusnih vzorcev

Bitumen and bituminous binders - Preparation of test samples

Bitumen und bitumenhaltige Bindemittel - Vorbereitung von Untersuchungsproben

Bitumes et liants bitumineux - Préparation des échantillons d'essai
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ICS:

75.140	Voski, bitumni in drugi naftni proizvodi	Waxes, bituminous materials and other petroleum products
91.100.50	Veziva. Tesnilni materiali	Binders. Sealing materials

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en

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English Version

Bitumen and bituminous binders - Preparation of test samples

Bitumes et liants bitumineux - Préparation des échantillons
d'essai

Bitumen und bitumenhaltige Bindemittel - Vorbereitung von
Untersuchungsproben

This European Standard was approved by CEN on 3 February 2007.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN 12594:2007) has been prepared by Technical Committee CEN/TC 336 "Bituminous binders", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2007, and conflicting national standards shall be withdrawn at the latest by September 2007.

This document supersedes EN 12594:1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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1 Scope

This European Standard specifies a method for preparing samples of bituminous binders in order to test their properties.

WARNING — Use of this European standard can involve hazardous materials, operations and equipment. This European standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this European standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced standard (including any amendments) applies.

EN 58, *Bitumen and bituminous binders - Sampling bituminous binders*

EN 1425, *Bitumen and bituminous binders - Characterization of perceptible properties*

EN 1427, *Bitumen and bituminous binders - Determination of the softening point - Ring and Ball method*

EN 1429, *Bitumen and bituminous binders - Determination of residue on sieving of bitumen emulsions, and determination of storage stability by sieving*

EN 1431, *Bitumen and bituminous binders - Determination of recovered binder and oil distillate from bitumen emulsions by distillation*

EN 12607-1, *Bitumen and bituminous binders - Determination of the resistance to hardening under the influence of heat and air - Part 1: RTFOT method*

EN 12607-2, *Bitumen and bituminous binders - Determination of the resistance to hardening under the influence of heat and air - Part 2: TFOT method*

EN 12607-3, *Bitumen and bituminous binders - Determination of the resistance to hardening under the influence of heat and air - Part 3: RFT method*

EN 12697-1, *Bituminous mixtures - Test methods for hot mix asphalt - Part 1: Soluble binder content*

EN 12697-2, *Bituminous mixtures - Test method for hot mix asphalt - Part 2: Determination of particle size distribution*

EN 12697-4, *Bituminous mixtures - Test methods for hot mix asphalt - Part 4: Bitumen recovery: Fractionating column*

EN 12847, *Bitumen and bituminous binders - Determination of settling tendency of bitumen emulsions*

EN 13074, *Bitumen and bituminous binders - Recovery of binder from bitumen emulsions by evaporation*

EN 14769, *Bitumen and bituminous binders - Accelerated long-term ageing conditioning by a Pressure Ageing Vessel (PAV)*

EN 14895, *Bitumen and bituminous binders - Stabilisation of binder from bituminous emulsions or from cut-back and fluxed bituminous binders*

EN ISO 3696:1995, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*

ISO 5280, *Xylene for industrial use – Specification*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 58 and the following apply.

3.1

laboratory sample

sample of bituminous binder intended for laboratory tests. It may be a spot sample, a composite sample or a part thereof (a divided sample)

3.2

test sample

sample of bituminous binder produced by treatment or subdivision of a laboratory sample for individual testing

4 Principle

In order to produce the laboratory sample the test material shall have been sampled in accordance with EN 58.

Perceptible properties of the test material shall be checked in accordance with EN 1425, prior to performing the present standard.

Homogenize the laboratory sample before the test samples are taken.

For certain tests (7.3.2) transfer the laboratory sample directly to the test sample containers and for other tests (7.3.3) sieve the laboratory sample prior to testing.

Store the laboratory sample from which the test samples are taken under controlled conditions. Stir gently to render it uniform and warm the sample, if necessary.

Prepare all test samples that are required for one property at the same time.

5 Reagents and materials

5.1 General

Use only reagents of standard analytical grade and water conforming to grade 3 of EN ISO 3696:1995.

5.2 Solution S_a

Aqueous solution with a nominal 0,01 mol/L of sodium hydroxide (NaOH) containing a nominal 1 % mass fraction sodium oleate. This solution may be used for preparing anionic emulsion samples. The actual soap solution may be used if available on site, in case of routine tests or for simplicity.

NOTE S_a stands for "anionic solution" as this solution is used for preparing anionic emulsion samples.

5.3 Solution S_c

Aqueous solution of nominal 0,01 mol/l of hydrochloric acid (HCl) containing a nominal 1 % mass fraction cetyltrimethylammonium bromide. This solution may be used for preparing cationic emulsion samples. The actual aqueous phase may be used if available on site, in case of routine tests or for simplicity.

NOTE S_C stands for "cationic solution" as this solution is used for preparing cationic emulsion samples.

5.4 Xylene, grade 2 conforming to ISO 5280.

5.5 Ethanol, 99 % volume fraction minimum or 95 % volume fraction, denatured.

6 Apparatus

Usual laboratory apparatus and glassware, together with the following:

6.1 Ventilated oven or ventilated laboratory oven with a temperature accuracy of ± 5 °C, checked at midpoint and working space at suitable intervals.

6.2 Indirect heating apparatus, e.g. hot jacket oven with thermometer, oil bath with thermometer or equivalent.

6.3 Melting pan of appropriate material

6.4 Any appropriate stirrer, e.g. manual (such as spatula) or mechanical equipped with a propeller.

6.5 Metal sieve, mesh size 0,50 mm, made from brass or stainless steel if used with bitumen emulsions.

6.6 Aluminium foil or lid for melting pan.

6.7 Test sample container or moulds of appropriate material with a lid or other closure, or glass conical flask with a ground glass stopper.

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7 Procedure for sample preparation

7.1 Solid or semi solid samples

7.1.1 Samples up to 1 litre

Ease the lid or other closure of the sample container and place the container with the lid loose in the oven for a maximum of 120 min at not more than 100 °C above the expected softening point as defined in EN 1427.

For modified bitumen use the same procedure if no other guidance is provided by the supplier. In any case, 200 °C shall not be exceeded.

Remove the container from the oven and stir (6.4) the melted sample with care in order to avoid incorporating air bubbles into the sample. For modified binders stir according to handling suggestion from supplier, when available. Allow any air bubbles to escape, if necessary by placing the sample in the oven for not more than 5 min. Pour the liquefied and homogenised sample into the moulds or test sample containers.

Carry out the entire procedure (melting, homogenising and moulding) within 135 min.

Discard the residue sample that has been heated.

7.1.2 Samples greater than 1 litre

If division of a sub-sample is necessary, ensure that the sub-sample is representative of the bulk sample. If necessary take a sufficient amount of material (100 g minimum) from the container by using an appropriate tool (e.g. a warmed but not glowing knife) and transfer the material into the melting pan.

Place the melting pan in the appropriate heating device (6.1 or 6.2).

Melt the material by stirring (6.4) at a maximum temperature not more than 100 °C above the expected softening point as defined in EN 1427. For modified bitumen use the same procedure, if no other guidance is provided by the supplier. In any case, 200 °C shall not be exceeded.

Lower temperatures required at later stages shall be achieved by cooling.

The whole sample shall be melted as follows:

- for 1 l to 2 l, 3 h maximum,
- for 2 l to 3 l, 3h 30 maximum,
- for 3 l to 5 l, 4 h maximum and
- for more than 5 l, overnight.

NOTE For samples larger than 5 l, the melting temperature of the material should be 50°C above the Ring and Ball softening point. As melting overnight is performed at a lower temperature, the temperature will be increased approximately 2 h before starting the sampling.

Stir the melting sample with care in order to prevent local overheating and avoid incorporating air bubbles into the sample. For a modified binder, stir according to the handling guidance provided by the supplier, if available. If the handling guidance provided by the supplier is unavailable, the modified bitumen shall be homogenised for up to 10 min, avoiding incorporating air bubbles into the sample. If necessary, cover the melting pan with aluminium foil or with a loose fitting lid.

Pour the liquefied and homogenised sample into the moulds or test sample containers.

Carry out the homogenising procedure and the pouring procedure within 15 min maximum.

Discard the residue sample that has been heated.

7.2 Soft bitumen, fluxed or cut-back binders

If necessary, to minimise the loss of volatiles during heating, cover the container with aluminium foil or a loose fitting lid. Homogenise the entire sample by gently stirring manually.

If the viscosity is too high, the sample shall be heated with care in a hot air or oil bath or equivalent for the minimum time required until it becomes sufficiently fluid to pour.

The bulk binder sample shall be placed in an oven maintained at a temperature which should not exceed whichever is the lower of the following temperatures:

- 80 °C above the estimated ring and ball softening point temperature;
- 140 °C for fluxed bituminous binders;
- 80 °C for cut-backs.

Avoid incorporating air bubbles into the sample by allowing any air bubbles to escape.